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**14. ABSTRACT**
Studies of the spectroscopy and dynamics of transient energetic species by anion photodetachment and cation recombination processes are reported. Experimental efforts focused on sulfur, nitrogen and oxygen clusters. The research included the first report of a 5-fold coincidence measurement in a study of $O_8^- \rightarrow O_2+O_2+O_2+O_2+e^-$

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Dissociative photodetachment, dissociative recombination, charge exchange, coincidence spectroscopy

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# Spectroscopy and Dynamics of Transient Energetic Species

## Annual Report - AFOSR Grant #F49620-000-10-010

## 12/1/99 – 11/30/2002

**April 30, 2003**

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## 1. Objectives

The objectives of the first half of this research project were to characterize the dynamics of neutral three-body dissociation reactions important in the atmosphere and to study the structure and dynamics of potential energetic materials of interest to the US Air Force. The experiments carried out to made use of a novel photoelectron-photofragment spectrometer constructed with the assistance of both the AFOSR and the David and Lucile Packard Foundation. This spectrometer employed a unique multiple photofragment detector to allow characterization of three-body dissociation processes. Negative-ion photodetachment was used to prepare transient neutral molecules and clusters as precursors to three-body dissociation reactions. The research focused on the three-body dissociative photodetachment of oxide and hydroxide ions solvated with water molecules and also continued earlier studies of nitrogen oxide species. In addition, experiments were carried out on the sulfur oxides $S_2O_2^-$ and $(SO_2)_3^-$. The oxide and hydroxide ions provide a route to studying the interactions of neutral O and OH radicals with water that may be of importance in modeling rocket exhaust signatures. Nitrogen and sulfur oxides are of general interest in the understanding of atmospheric chemistry, and one of the nitrogen oxides we studied, $N_2O_2$, has been previously proposed as a candidate for an energetic material, however we found no evidence for stable high-energy isomers. A final application of this multiparticle spectrometer to many-body dissociative photodetachment in a study of $O_8^- + h\nu \rightarrow O_2 + O_2 + O_2 + O_2 + e^-$ was reported, constituting one of the highest-dimension coincidence experiments ever carried out in chemistry.

In the latter half of the grant period, the project moved in a new direction: studies of the dynamics and product channels in the dissociative recombination (DR) of electrons with small hydrocarbon ions, an area in which information is needed to model ion-assisted combustion processes important to AF interests. Initial efforts to carry out DR studies by interaction of a cation beam with a nearly stationary cloud of electrons formed in a reversal electron gun failed to yield results, owing to the low number density of electrons established. These experiments were followed by an effort to study dissociative charge-exchange with Rydberg rare gas atoms, however these experiments were also complicated by low target number densities and competing ionization processes in the Rydberg rare gas atom source. Finally in the last months of the grant, successful experiments on the charge exchange and collision-induced dissociation of cations in collisions with NO and Cs targets began to produce results.

## 2. Accomplishments/ Findings 12/1/99 – 11/30/02

### 2.a. Dissociative Photodetachment Studies of $O^-(H_2O)_2$, $OH^-(H_2O)_2$

In these studies, photoelectron-photofragment coincidence (PPC) spectroscopy was used to study dissociative photodetachment (DPD) of the doubly hydrated clusters of oxide and hydroxide, $M^-(H_2O)_2 \rightarrow M + 2H_2O + e^-$ where M = O and OH. These experiments yield information on the energetics of the parent anion and the dissociation dynamics of the photodetached neutral species. Photoelectron and PPC spectra are measured and compared to data for $O^-(H_2O)$ and $OH^-(H_2O)$. Unlike the singly hydrated species, no evidence of vibrationally resolved product translational energy distributions was observed. The second hydration energy of $O^-$ with both $H_2O$ and $D_2O$ was also measured to be $0.80 \pm 0.08$ and $0.81 \pm 0.08$ eV, respectively.

The three-body dissociation dynamics of the neutral clusters produced by photodetachment were studied by measuring the velocities and recoil angles of all the particles in coincidence. The observed partitioning of momentum was found to be consistent with a two-step mechanism or dissociation from a wide range of starting geometries. In future efforts it will be interesting to photodetach at higher photon energies to determine if electronically excited O or OH have an effect on the three-body dynamics. *(publication 5)*

**2.b. Studies of the Excited State Dynamics of $N_2O_2$ by Dissociative Photodetachment of $N_2O_2^-$.**

We have also completed our studies of the excited states of $N_2O_2$. These have been characterized by experiments on the DPD of $N_2O_2^-$ at 266 nm. In this case, PPC spectroscopy is used to uniquely reveal the correlation between features observed in the photoelectron spectrum and different neutral dissociation pathways. Evidence for at least two isomers of $N_2O_2^-$ was found and upper limits for their stabilities were determined. One, $ONNO^-$, is stable relative to NO + NO + $e^-$ by $< 1.70 \pm 0.05$ eV, while the second one, trigonal $N_2O_2^-$, is stable relative to $O^-$ + $N_2O$ by $< 0.57 \pm 0.05$ eV. The observed dissociation channels are assigned to $ONNO \rightarrow NO + NO$, $N_2O_2 \rightarrow O\ (^3P) + N_2O$ and either $O(^1D) + N_2O$ or $N(^4S) + NO_2$. No evidence for stable $N_2O_2$ was found in these experiments, showing that the low-lying excited states, at least those accessible by photodetachment of these relatively strongly bound anions, are not good candidates for energetic materials. *(publication 7)*

**2.c. Dissociative Photodetachment Dynamics of $S_2O_2^-$.**

In this work, PPC spectroscopy was employed to study the dissociative photodetachment of $S_2O_2^-$ at 258 nm. This represents the first experimental study of this anion. The experimental data and theoretical calculations show evidence for photodetachment from a trigonal form of $S_2O_2^-$. The vertical detachment energy of this isomer was determined to be $3.72 \pm 0.02$ eV. An upper bound of $2.41 \pm 0.06$ eV is determined for the enthalpy of the reaction $S_2O_2^- \rightarrow S + SO_2 + e^-$ at 0K. The observed dynamics are interpreted in terms of dissociative photodetachment of $S_2O_2^-$ to $S(^3P) + SO_2(^1A_1) + e^-$, $S(^1D) + SO_2(^1A_1) + e^-$, and $S_2(^3\Sigma_g^-) + O_2(^3\Sigma_g^-) + e^-$ product channels. The S-atom channels are characterized by a large photofragment kinetic energy release and an anisotropic photofragment angular distribution peaked along the electric vector of the laser. The $S_2$ channel has a low kinetic energy release consistent with elimination of highly vibrationally excited $O_2$ from a strained form of the trigonal isomer. *(publication 6)*

**2.d. Effects of clustering NO with $NO_2^-$: $N_2O_3^-$ and $N_3O_4^-$**

Nitrogen oxides are important both as environmental pollutants and potential energetic materials. In these experiments the energetics and DPD dynamics of $N_2O_3^-$ and $N_3O_4^-$ cluster anions were studied. These systems can be thought of as $NO_2^-(NO)$ and $NO_2^-(NO)_2$ given that the electron affinity of $NO_2$ is much higher than that of NO. The $N_2O_3^-$ spectrum largely reproduces the photoelectron spectrum (with a 0.28 eV shift in the electron affinity) of $NO_2^-$, including the observation of photoelectron bands corresponding to detachment to the ground state but also the first two excited electronic states of $NO_2$. The $N_3O_4^-$ spectrum, however,

exhibits a significant quenching of the excited state signals – signal corresponding to the first excited *B* state of $NO_2$, for example, is reduced by a factor of three relative to $N_2O_3^-$. This may indicate that $N_3O_4^-$ is not a simple cluster anion and may exhibit several isomers, as seen in our earlier work on $N_3O_2^-$. Solvation of $NO_2^-$ by one NO does not significantly affect the energetics and geometry of the anion, while addition of a second NO alters the geometry of the $NO_2^-$. As alluded to above, this effect leads to a suppression in the photodetachment cross section for the $a^2B_2$ excited state of $NO_2$ in the $NO_2(NO)_2$. The PPC spectra also suggested that the three-body dissociation process of the $NO_2(NO)_2$ occurs via a sequential decay mechanism. *(publication 8)*

**2.e. Three-Body Dissociation Dynamics: $(SO_2)_3^-$**

Sulfur oxides are important participants in the chemistry of atmospheric pollution, particularly the formation of acid rain. PPC spectroscopy was used to study the energetics and dissociation dynamics of the neutral $(SO_2)_3$ produced by photodetachment of the trimer anion. The dissociation dynamics of $(SO_2)_2$ were previously studied in this laboratory and it was determined that the dissociation dynamics were most consistent with the presence of an S-O bond between the two $SO_2$ moieties. In the trimer there is evidence that the $(SO_2)_2$ core remains, and the third $SO_2$ acts essentially as a spectator. Using both molecular-frame differential cross-sections and Dalitz representations of the momentum partitioning in this system, it can clearly be seen that most of the momentum is imparted to two $SO_2$ molecules, and the third molecule receives little momentum, confirming that dissociation is occurring with a dimer core and a spectator $SO_2$. *(publication 10)*

**2.f. Four-Body Dissociation Dynamics: Dissociative Photodetachment of $O_8^-$**

In the final experiments done before converting the apparatus to study dissociative recombination processes, we extended the PPC technique to a four-body DPD process: the four-body DPD of $O_8^-$. Using our unique quad crossed-delay-line detector, we were able to detect all four neutral $O_2$ fragments that arise from the DPD of $O_8^-$ in coincidence with the photodetached electron, providing a kinematically complete 5-body experiment. From these experiments, insight into the energetics and 4-body dissociation dynamics of the $O_8$ neutral is obtained. The observed velocity and angular correlations for DPD of $O_8^-$ are found to be similar to those of $O_4^-$ and $O_6^-$ implying that the additional solvating $O_2$ molecules act essentially as spectators, but exhibit inequivalent kinematic behavior implying asymmetric solvation. *(publication 9)*

**2.g. Dissociation Dynamics in Homogeneous $NO_2$ clusters**

Anionic homogeneous clusters of nitrogen dioxide, $(NO_2)_{n=1-5}^-$, were studied by dissociative photodetachment (DPD) at 258 nm. Insight into the structures of these clusters was obtained through photoelectron and photofragment translational spectroscopy. Both $(NO_2)_2^-$ and $(NO_2)_3^-$ undergo two-body DPD even though three-body DPD is energetically accessible for the trimer, implying a change in cluster geometry between the dimer and trimer. This change is also reflected in the photoelectron spectra, where the ground state peak narrows and undergoes a shift of 0.34 eV upon addition of a third $NO_2$ molecule while the peak shift from the monomer to the dimer is only 0.22 eV. The larger shift for the addition of the third $NO_2$ is in contrast to incremental solvation shifts in most clusters that monotonically decrease with increasing cluster size. The photoelectron spectra of $(NO_2)_4^-$ and $(NO_2)_5^-$ are consistent with the spectrum of

$(NO_2)_3^-$, and likely arise from the presence of an $N_2O_4^-$ core which does not dissociate upon photodetachment. This work is still in preparation for publication.

### 2.h. Dissociative Recombination and Charge Exchange Processes

Our initial efforts to study electron-cation recombination dynamics, initiated in September, 2001, focused on using our pulsed fast ion beam technology in a variant of classic merged beam experiments involving the interaction of a keV ion beam with a nearly stationary cloud of electrons. The main challenge in these studies was the design and implementation of the low energy electron source using a reflection electron gun. This experimental approach involves reversing a several hundred eV electron beam in a shaped potential wall. This approach has been shown by Chutjian, et al. and Wodtke, et al. to produce a number of negative ions from gas cells and molecular beams with attachment resonances near 0 eV. After a number of experimental attempts, including direct imaging of the electron beam and construction of a second generation electron gun with a much longer (1 cm) interaction region, however, this approach has been abandoned. The fundamental problem with this approach is the space charge in the electron reversal region, a problem that is reduced at the higher pressures used in electron-attachment mass spectrometry on gases and molecular beams.

Our primary goal remains the direct experimental study of the recombination processes of free electrons and polyatomic cations, providing complementary data and extending this data to larger polyatomic cations than is possible with the only competitive approach – dissociative recombination experiments on ion storage rings in Sweden, Denmark and Germany. As we tackle this tought problem, we have set up an interim experiment to study charge exchange and collision-induced dissociation processes. The first effort involved the interaction of cations with high-n Rydberg rare gas atoms in a crossed beam configuration. This approach, using an electron-impact excited rare gas beam, allowed us to create a reasonable number density of highly excited rare gas atoms without the space-charge limitations inherent in an electron beam. The goal here was to deliver near-zero-energy electrons to the pulsed cation beam and to find scattering cell conditions with minimal hard collisions between the cations and the rare-gas beam, but an acceptable number of long-range charge-transfer recombination collisions producing the highly excited neutral species we have set out to study, including hydrocarbon and cluster cations. This effort was plagued by problems with target number density in the skimmed, differentially pumped rare gas beam and also a large background of cations from the electron-impact Rydberg atom source.

In the last weeks of the grant we converted the Rydberg atom experiment to a charge-exchange / collision-induced dissociation configuration in which the pulsed cation beam interacted with collision partners in a small, relatively high-pressure gas cell. Initial results on the two- and three-body dissociative charge-exchange processes of $(O_2)_n^+$ and $(N_2)_n^+$ clusters with permanent gases included $O_2$, $N_2$, Ar and NO were obtained. This work is now continuing under the renewal of this research grant and will be reported on in the future.

### 3. Personnel Associated with the Research Effort

1. A number of personnel have received full or partial support from this grant:

Graduate Students: Leah Alconcel, Todd Clements, Chris Laperle and A. Khai Luong

A. Khai Luong, Todd Clements and Leah Alconcel all received the Ph.D. degree during the period of this grant and have gone on to research positions at other institutions. Dr. A. Khai Luong is a staff scientist at the Combustion Research Facility at Sandia National Laboratory, Dr. Todd Clements is a postdoc at the University of Christchurch in New Zealand and Dr. Leah Alconcel is an NRC postdoctoral fellow at JPL/Caltech.

2. Other personnel have worked on these projects with other AFOSR support:

Graduate Students: A. Khai Luong (AASERT), Todd Clements (AASERT), Chris Laperle (AASERT)

Undergraduate Students: Raymond Liu (AASERT)

No postdoctoral fellows have been supported on grant during this time, but Dr. Hans-Juergen Deyerl did some work on AFOSR projects supported by a Deutsche Forschungsgemeinschaft stipend.

**4. Publications**

In the period of this grant, AFOSR supported research has been published as enumerated here.


1. R.E. Continetti, "Dissociative Photodetachment Studies of Transient Molecules by Coincidence Techniques.", pp. 748-808 in Advanced Series in Physical Chemistry: Photoionization and Photodetachment, ed. C.Y. Ng, World Scientific, Singapore (2000).
2. A.K. Luong, T.G. Clements and R.E. Continetti, "Three-body Dissociation Dynamics of Neutral Transient Species: Dissociative Photodetachment of $O_6^-$.", ACS Symposium Series Vol. 770, Imaging in Chemical Dynamics, eds. A.G. Suits and R.E. Continetti, ACS, Washington DC (2000), pp. 312-325.
3. A.G. Suits and R.E. Continetti, "Imaging in Chemical Dynamics - the State of the Art.", ACS Symposium Series Vol. 770, Imaging in Chemical Dynamics, eds. A.G. Suits and R.E. Continetti, ACS , Washington DC (2000), pp. 1-18.
4. R.E. Continetti, "Coincidence Spectroscopy.", Annual Reviews of Physical Chemistry, Vol. 52, (2001), pp. 165-192.
5. T.G. Clements, A.K. Luong, H.-J. Deyerl and R.E. Continetti, "Dissociative Photodetachment Studies of $O^-(H_2O)_2$, $OH^-(H_2O)_2$ and the Deuterated Isotopomers: Energetics and Three Body Dissociation Dynamics.", J. Chem. Phys. **114**, 8436-8444 (2001).
6. T.G. Clements, H.-J. Deyerl and R.E. Continetti, "Dissociative Photodetachment Dynamics of $S_2O_2^-$.", J. Phys. Chem. A. **106**, 279-284 (2002).
7. R.-J. Li and R.E. Continetti, "Studies of the Excited State Dynamics of $N_2O_2$ by Dissociative Photodetachment of $N_2O_2^-$.", J. Phys. Chem. A. **106**, 1183-1189 (2002).
8. A.K. Luong, T.G. Clements and R.E. Continetti, "Photoelectron-Photofragment Coincidence Spectroscopy of $NO_2^-(NO)_{1,2}$: Solvation Effects of NO on $NO_2^-$.", Intl. Journal of Mass Spectrometery **220**, 253-262 (2002).

9. T.G. Clements and R.E. Continetti, "Four-body Reaction Dynamics: Complete Correlated Fragment Measurement of the Dissociative Photodetachment Dynamics of $O_8^-$.", Phys. Rev. Lett. **89**, 033005-1 – 033005-4 (2002).
10. T.G. Clements, A.K. Luong and R.E. Continetti, "Three-body dissociation dynamics of $(SO_2)_3$ studied by dissociative photodetachment of $(SO_2)_3^-$.", Chem. Phys. Lett. **366**, 650-655 (2002).

## 5. Interactions/Transitions

### 5.a. Meetings

The P.I. gave several talks on AFOSR sponsored research during this grant, detailed here:

1. "Coincidence Studies of the Dynamics of Transient Species.", 3rd Meeting of the Birmingham Centre for Chemical Physics: "Reaction Dynamics in the Gas-Phase, in Clusters and on Surfaces.", University of Birmingham, UK, April 6, 2000.
2. "Dissociative Photodetachment Studies of Transient Species.", Department of Chemistry, University of Rome La Sapienza, Rome, Italy, April 13, 2000.
3. "Dissociative Photodetachment Studies of Transient Species and Three-Body Dissociation Dynamics.", Department of Chemistry, University of Rome La Sapienza, Rome, Italy, May 16, 2000.
4. "Spectroscopy and dynamics of transient species," AFOSR Contractor's Meeting, Waltham, MA, May 22, 2000.
5. "Energetics and Dynamics of Clusters of Oxygen: $O_4^-$ and $O_6^-$.", CNR Institute of Chemistry and Thermodynamics at High Temperatures, University of Rome La Sapienza, Rome, Italy, June 14, 2000.
6. "Dissociative Photodetachment Studies of Transient Species.", LCAM, CNRS, Universite Paris Sud, June 28, 2000.
7. "Multi-particle Detection Techniques for Chemical Dynamics.", IMAGINE Network Meeting, Heraklion, Greece, Oct. 23, 2000.
8. "Pulsed Negative-Ion-Beam Photoelectron Spectroscopy using Imaging Techniques, I, II, III and IV.", Laboratory of Laser Photochemistry and Dynamics, Department of Chemistry, University of Rome La Sapienza, Rome, Italy, November 2,3,6 and 7, 2000.
9. "Studies of Three-Body Dissociation Dynamics by Dissociative Photodetachment.",Gordon Research Conference on Molecular Energy Transfer, Ventura, CA, January 15, 2001.
10. "Dissociative Photodetachment of Cluster Anions: Insights into Three-Body Dissociation Dynamics.", Western Spectroscopy Association Meeting, Feb. 1, 2001.
11. "Dissociative Photodetachment Dynamics.", Symposium on Molecular Photoelectron Spectroscopy, 221st ACS National Meeting, San Diego, CA April 2, 2001.
12. "Fast-Beam Coincidence Imaging Studies of Negative-Ion Photodetachment.", Gordon Research Conference on Photoions, Photoionization and Photodetachment, Williams, Massachusetts, July 11, 2001
13. "Transition State Dynamics and Three-Body Collisions.", 18th Conference on the Dynamics of Molecular Collisions, Copper Mountain, Colorado July 16, 2001.

14. "Anion Dissociation Dynamics by Photoelectron-Photofragment Coincidence Studies.", 22nd International Conference on Photonic, Electronic and Atomic Collisions, Santa Fe, New Mexico, July 19, 2001.
15. "Many-body Dissociation Dynamics and Photoelectron-Photofragment Coincidence Spectroscopy.", Max Born Institute, Berlin, Germany, August 27, 2001.
16. "Studies of Transient Species by Photoelectron-Photofragment Coincidence Spectroscopy.", Institute of Physics, University of Kaiserslautern, Kaiserslautern, Germany, August 28, 2001.
17. "Studies of Transient Species by Photoelectron-Photofragment Coincidence Spectroscopy.", Institute of Physical Chemistry, University of Karlsruhe, Karlsruhe, Germany, August 30, 2001.
18. "Transition States and Three-Body Collisions.", University of Nevada, Reno, Department of Chemistry, November 2, 2001.
19. "Spectroscopy and Dynamics of Transient Energetic Species and Dissociative Recombination Dynamics of Molecular Cations.", AFOSR Contractor's Meeting, May 20, 2002.
20. "Many Body Dissociation Dynamics in Dissociative Photodetachment and Charge Exchange Processes," Departmental Colloquium, The Johns Hopkins University, April 22, 2003.

In addition to these presentation at meetings, the PI was a co-organizer of a scientific symposium during the period of this grant, and has also been elected vice-chair of the 2003 Gordon conference on Photoions, Photionization and Photodetachment, Oxford, UK Sept. 21 – 26, 2003:

Co-organizer, Pacifichem 2000 Symposium, "New Frontiers in Chemical Reaction Dynamics", Honolulu, Hawaii, December 14-19, 2000.

Vice-Chair, Gordon Conference on Photoions, Photoionization and Photodetachment, Oxford, UK September, 2003.

The PI's group also presented the following posters focusing on AFOSR-funded research results:

1. T.G. Clements, A.K. Luong, H.-J. Deyerl and R.E. Continetti, "Energetics and Three Body Dissociation Dynamics of $O^-(H_2O)_2$, $OH^-(H_2O)_2$ and Their Deuterated Isotopomers.", Conference on Molecular and Ionic Clusters, Toulouse, France, April 16-20, 2000. (Poster)
2. A.K. Luong, T.G. Clements and R.E. Continetti, "Dissociation Dynamics of Small Water Cluster Anions.", Symposium on New Frontiers in Reaction Dynamics, Pacifichem 2000, Honolulu, HI Dec. 14-19, 2000. (Contributed Talk) (Poster)
3. T.G. Clements, A.K. Luong and R.E. Continetti, "Dissociative Photodetachment Dynamics of the Sulfur Oxides $(SO_2)_3^-$ and $SO_3^-(H_2O)$.", 221st ACS National Meeting, San Diego, CA April 4, 2001. (Poster)
4. R.E. Continetti, "Spectroscopy and Dynamics of Transient Energetic Species.", AFOSR Molecular Dynamics Contractor's Meeting, Irvine, CA May 21, 2001. (Poster)

**5.b. Consultative/Advisory Functions**

During this grant period, the PI attended and presented talks at the AFOSR contractors meeting in Waltham, Mass, May 2000 and May 2002. In addition, the PI engaged in a number of discussions with AFRL researchers Dr. Robert Morris, Dr. Skip Williams, Dr. Al Viggiano and Dr. Susan Arnold concerning the potential for studying the dynamics of three-body dissociative

recombination processes in the laboratory at UCSD. The PI also discussed this work with Prof. Mats Larsson of Stockholm University, who carries out studies of absolute cross-sections for dissociative recombination at the CRYRING ion storage ring in Stockholm. These interactions have resulted in a new focus in our laboratory on studying the dynamics of dissociative recombination and charge-exchange processes.

**5.c. Transitions**

The P.I. has continued a collaboration with Dr. Carl Hayden at the Combustion Research Facility at Sandia National Laboratories. From January – March 2000, the PI worked in the laboratory of Dr. Hayden while on sabbatical, focusing on studies of transient hydrocarbon radicals with ultrafast laser techniques. The PI also spent April – June 2000 at the University of Rome, La Sapienza in the laboratory of Prof. Anna Giardini. In that period, studies of laser ablation techniques for the production of anions were carried out and the construction of a photoelectron spectrometer was commenced.

## 6. New Discoveries, Inventions or Patent Disclosures

Outside of the results reported in the accomplishments/new findings section above, there is nothing further to report here.

## 7. Honors/Awards

In the period of this grant, the principal investigator, Prof. Robert E. Continetti was elected to be a Fellow of the American Physical Society.